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# *X-RAY DIFFRACTION MICROSCOPY*

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**Summary:** X-ray diffraction microscopy enables fast 3D mapping of embedded grains, domains and in some cases dislocations within mm-sized specimens. The spatial and angular resolution is variable down to 50 nm and 0.001°, respectively. The methodology is presented with a view to multiscale modelling and applications from plasticity, recrystallization, multiferroics, fuel cell research and bio-minerals.

## 1. INTRODUCTION

Most crystalline materials, including metals, ceramics, rocks and bio-minerals, exhibit a structure that is organized hierarchically, e.g. in grains, domains, and atomic-scale defect networks. A critical issue across materials and geological sciences is to understand and model the interplay of physical phenomena and structural dynamics at and between these different length scales. With this capability, the final macroscopic properties of a material can then be predicted from the initial microstructure and processing conditions, and materials can be designed and optimized using computer simulations. To guide such efforts, it is vital to develop experimental techniques that can rapidly swap between different length scales and make in situ, three-dimensional (3D) movies of the changes in local structure, strain, and orientation.

X-ray diffraction microscopy is a new full-field imaging technique for nondestructively mapping structure, orientation, and strain within millimeter-sized samples in 3D [1,2]. By placing an x-ray objective in the diffracted beam from a given grain or domain embedded within a larger sample, one can zoom in on such an object. The objective also filters diffraction signals from other grains/ domains, thereby suppressing unwanted overlap and isolating the structural element of interest. The microscopy technique is readily combined with coarse-scale 3D grain-mapping techniques, such as 3D x-ray diffraction (3DXRD) and diffraction contrast tomography (DCT), as well as classical tomography and diffraction topography. This combination enables the user to progress from fast overviews of an entire specimen to detailed studies of local phenomena.

## 2. EXPERIMENTAL METHOD

The x-ray objective creates a magnified image of a specific grains or domain with a magnification of up to 50. Maps of axial strain can be obtained by scanning the objective and detector through the scattering angle $2\theta$, while the local misorientation can be mapped by scanning the sample through two orthogonal tilt directions ($\chi$ , $\phi$ ). Three-dimensional measurements of axial strain and orientation can be obtained in two ways: First, by using a one-dimensionally focusing condenser to create a narrow line beam that illuminates a “slice” of the material, which is then imaged at an oblique angle. A 3D volume is then obtained layer-by-layer by translating the sample through the planar beam in small increments. A second, faster method involves illuminating the entire grain and recording projections from different viewing angles while rotating the sample around the scattering vector. 3D reconstruction can then be accomplished using adapted tomographic algorithms.

A dedicated setup for x-ray diffraction microscopy has been established at beamline ID06 at the ESRF, operating in the x-ray energy range of 15–35 keV. The majority of the experiments have used a compound refractive lens as objective. In this case, the spatial resolution is ≈100 nm, the field of view 100 μm, and the strain resolution better than $10^{-4}$, while

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exposure times are of order 1 second. The numerical aperture (NA) is $10^{-3}$, implying angular resolutions of $0.001^{o}$ to $0.03^{o}$, superior to those of electron microscopes. Recently, in collaboration with S. Bajt from DESY we have demonstrated the use of Multilayer Laue Lenses (MLL) [3] as an alternative type of objective. The NA can be 5 times larger. At the time of writing 50 nm resolution has been demonstrated while a test for 25 nm is being prepared.

## 3. RESULTS

Results from selected applications are reproduced in Fig 1:

*a) Multiscale mapping of grains and domains in Al*.

*b) Processing of plastically deformed meals*. Shown are dislocations within a nucleus appearing during recystallisation of Al. These dislocations pin the growth of the nucleus

*c) Domain evolution in ferroelectrics*. Shown is a map of the axial strain within one layer of a deeply embedded 40 µm grain in $BaTiO_3$.

*d) Strain field around dislocations* in the interior of a diamond crystal. The colour indicates the strain.

e) *Bio-minerals*. Shown is a rocking curve scan of mussel shell nacre tablets with a brick-and-mortar-like structure showing a range of orientations around the growth axis, between and within different tablets.

### References

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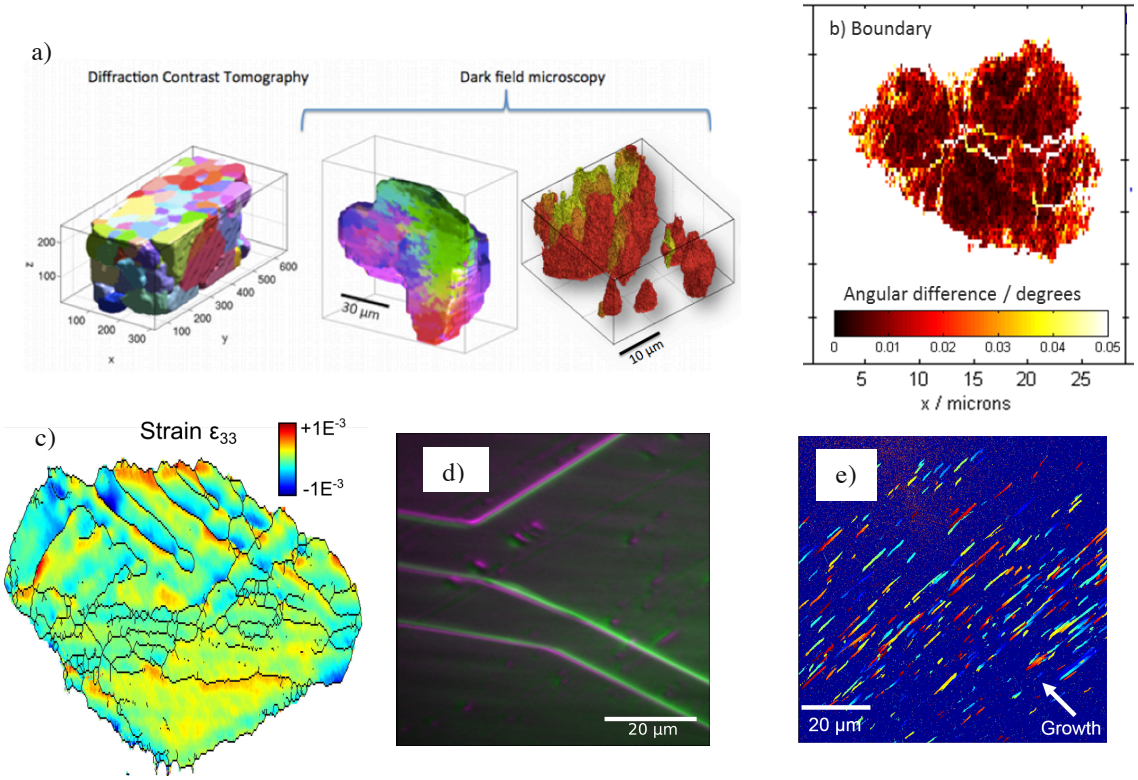


**Figure 1:** (a)-(e): Applications of x-ray diffraction microscopy, see main text.